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APPLICATION OF  
A RADIO-TRACER METHOD TO  
THE DECOMPOSITION MECHANISM OF  
POLYURETHANE PROPELLANT SYSTEMS (U)



U.S. NAVAL PROPELLANT PLANT INDIAN HEAD MARYLAND

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**RESEARCH AND DEVELOPMENT DEPARTMENT**

**NavWeps Report 8021  
Technical Report 121**

**16 November 1962**

**APPLICATION OF A RADIO-TRACER METHOD TO  
THE DECOMPOSITION MECHANISM OF POLYURETHANE  
PROPELLANT SYSTEMS(U)**

**By**

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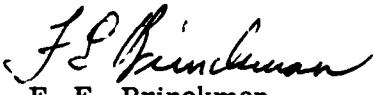
**JOE L. BROWNING  
Technical Director**

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## FOREWORD

The Naval Propellant Plant has been conducting research investigations for the past several years to improve methods of predicting the useful life of solid propellants. The experiments described in this report indicate that the radioactive tracer technique provides a convenient tool for following the degradation of polyurethane propellants. The data are as of 1 October 1962 and are subject to modification or withdrawal.

The names of manufacturers are given to identify types of material and equipment and do not constitute an endorsement of the products.



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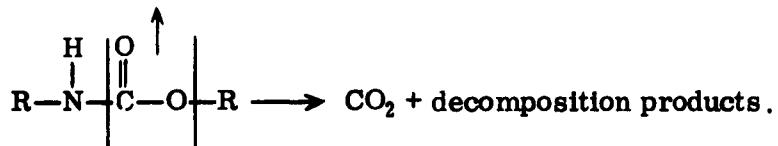
**Abstract Confidential****ABSTRACT**

A new technique employing radio-tracer C<sup>14</sup> has been used for following the thermal degradation of a polyurethane propellant. The described tracer method is thought to be an effective tool to investigate the influence of such parameters as moisture, pressure, other ingredients of the propellant, etc., on the rate of carbon dioxide evolution and, therefore, the rate of binder breakdown. Extrapolation of the data obtained indicates that the half life of the propellant is 6.7 years.

**APPLICATION OF A RADIO-TRACER METHOD TO  
THE DECOMPOSITION MECHANISM OF POLYURETHANE  
PROPELLANT SYSTEMS(U)**

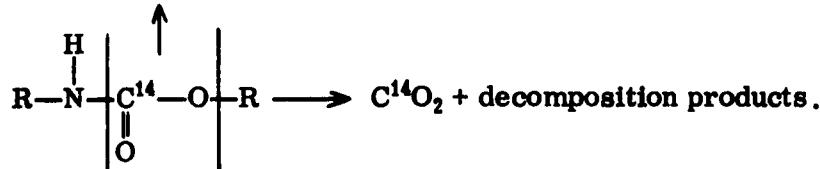
Several techniques have been employed for following the decomposition of polyurethane propellants in order to predict their safe, useful lives. Among the techniques used are stress relaxation measurements,(1,2) swelling measurements,(1) nuclear magnetic resonance measurements,(3) and extraction procedures.(4,5)

One paper(6) reports the evolution of gases consisting chiefly of carbon dioxide and hydrogen during thermal decomposition of a polyurethane propellant. It is believed that the urethane linkage of the propellant binder is one source for the CO<sub>2</sub> being evolved, according to



The purpose of the work described in this paper is (1) to determine unequivocally whether or not the urethane linkage is mechanistically involved in storage decomposition of this selected propellant, and (2) to evaluate a sensitive method for determining the rates of such decomposition. Furthermore, it was planned to investigate the rate of CO<sub>2</sub> evolution which is a measure of the rate of decomposition of urethane linkages; and therefore, also, a measure of decomposition of the propellant binder.

The approach in this problem involves using a radio-tracer method; i. e., labeling the carbon atom of the urethane linkage with radioactive C<sup>14</sup>,



It is obvious that if the urethane linkage breaks, C<sup>14</sup>O<sub>2</sub> should be evolved which can be easily detected by its radioactivity. This, in turn, is a demonstration for the decomposition of the urethane linkage.

As an example of a polyurethane propellant, the formulation of the Aerojet-General Polaris urethane propellant (ANP 2639 AF) was used. The basic ingredients of this propellant are ammonium perchlorate, 60%; aluminum, 15%; and a polyurethane binder, 24%.

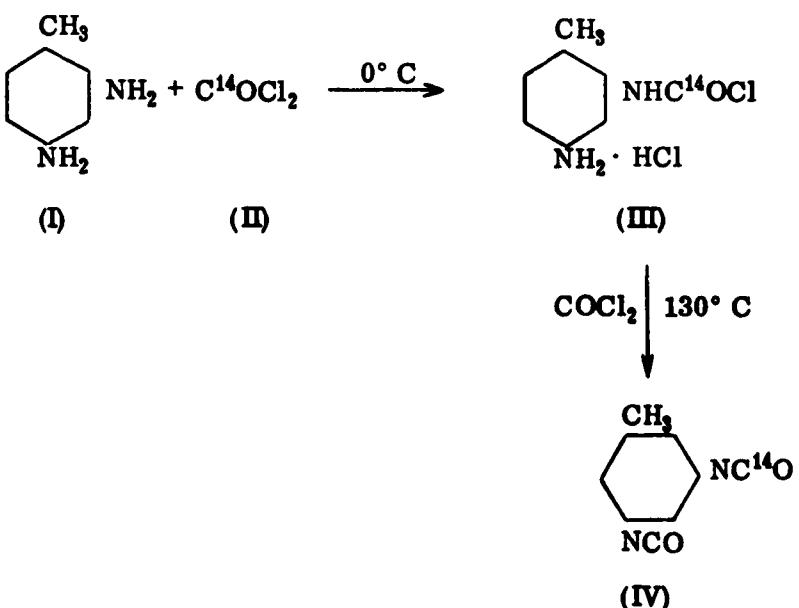
The binder itself consists of a three-dimensional network, made up of a 2,4-toluenediisocyanate (TDI)-polypropylene glycol (PPG) chain, crosslinked with monohydroxyethyl-tris-hydroxypropyl N,N,N',N'-ethylenediamine (MTDA).

The results obtained by employing this propellant system to study the decomposition of its urethane binder linkage by the radio-tracer method are described in the following sections.

## EXPERIMENTAL

### Preparation of Toluenediisocyanate Labeled with C<sup>14</sup>

In order to prepare a propellant which is labeled with C<sup>14</sup> at the urethane linkage of its binder, TDI labeled in one or both of its isocyanate groups is needed. Because it appears very difficult to label both isocyanate groups in a well defined way, TDI labeled in only one isocyanate group was prepared. The method reported by Siefken<sup>(7)</sup> for the preparation of isocyanates in general was employed. This method and the apparatus used were modified to meet the special requirements in synthesizing the desired labeled compound. The preparation of the labeled TDI is a two-step reaction as follows:



Toluenediamine (TDA, I) dissolved in methylene chloride is reacted at 0° C with labeled phosgene<sup>1</sup> which is also dissolved in methylene chloride. The intermediate compound (III) precipitates and is completely freed of methylene chloride and excess phosgene by evacuation.

In the second reaction step, compound III is suspended in high-boiling Aroclor 1248<sup>2</sup> and is reacted with unlabeled phosgene at 130° C until a clear solution is obtained. Finally, the labeled TDI is recovered from this solution by vacuum distillation. The apparatus shown in Figure 1 was used in this procedure.

Two batches of radioactive TDI were prepared by the following procedure:

For batch no. 1, 3.66 g of TDA (recrystallized from benzene) dissolved in 45 ml methylene chloride was reacted with 2.7 ml liquid phosgene carrier plus 0.1 millicurie of C<sup>14</sup>-phosgene (specific activity 1.52 millicuries per millimol) dissolved in 15 ml methylene chloride. The intermediate salt precipitate was freed from phosgene and methylene chloride, suspended in Aroclor, and reacted with phosgene gas until a clear solution was obtained. The TDI was recovered by vacuum distillation and analyzed for purity by reacting a sample with di-n-butylamine, and titrating the excess amine with hydrochloric acid. (8)

Batch no. 2 was prepared in the same way as batch no. 1 except 0.5 millicurie (specific activity 1.52 millicuries per millimol) radioactive phosgene was used.

Data of batch no. 1 and batch no. 2 are given below:

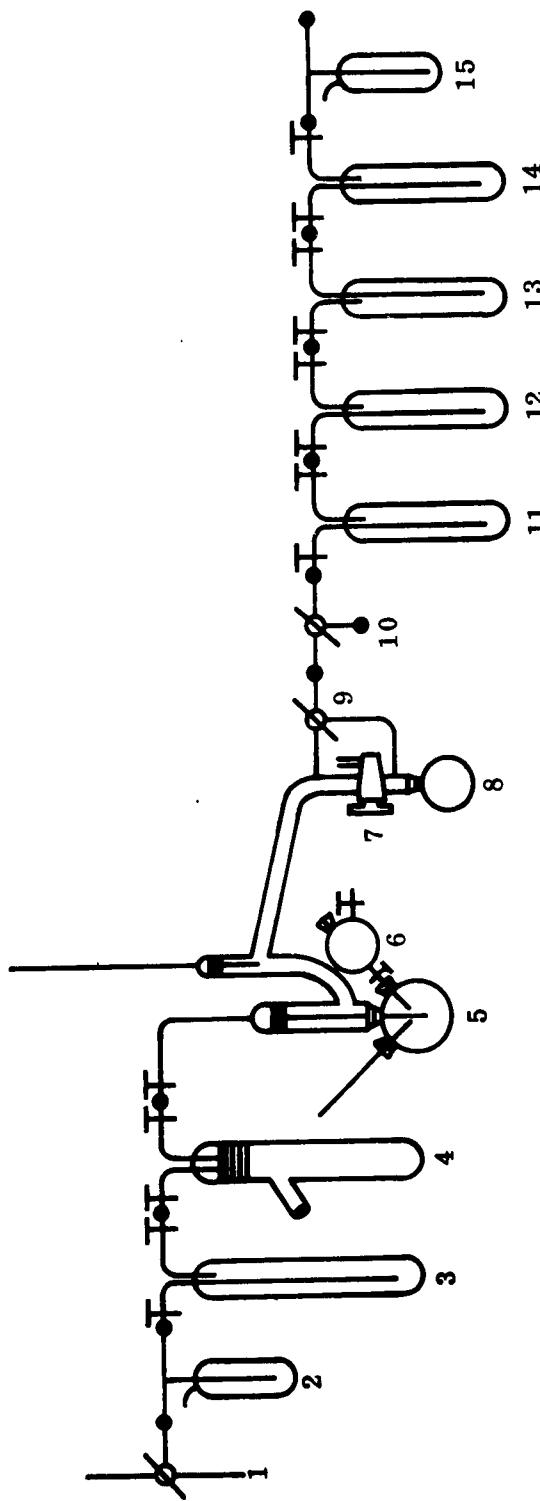
	<u>Batch no. 1</u>	<u>Batch no. 2</u>
Yield:	3.0 g TDI (57% of the theory)	3.0 g TDI (57% of the theory)
Boiling point:	90° C/2 mm Hg	90° C/2 mm Hg
Melting point:	21.3° - 21.6° C	20.8° - 21.3° C
Purity:	94.7%	97.8% (after two distillations)
Specific activity:	14.8 microcuries/gram TDI	51.0 microcuries/gram TDI
Total activity:	44.4 microcuries (44% of applied amount)	153 microcuries (30% of applied amount)

In order to obtain a purer product, 4 ml of inactive TDI (purity 99%) was added to the 3 g of labeled TDI (Batch no. 2) and the mixture distilled again. Results were as follows:

Yield:	5.74 g TDI
Boiling point:	90° C/2 mm Hg
Melting point:	20.8° - 21.5° C
Purity:	98.6%
Specific activity:	21.0 microcuries/gram

<sup>1</sup>Obtained from Nichem Company.

<sup>2</sup>Obtained from Monsanto Chemical Company.



1. Three-way stopcock to admit either phosgene or nitrogen gas.
2. Stock valve.
3. Trap to condense unlabeled phosgene as a carrier.
4. Trap to open ampoule containing labeled phosgene (by means of steel ball and magnet) and to add non-radioactive phosgene carrier.
5. Pyrex flask (50-ml) in which a solution of phosgene in methylene chloride is prepared and reacted with TDI solution in methylene chloride admitted through dropping funnel (6).
6. Dropping funnel.
7. Two-way stopcock.
8. Receiving flask.
9. Three-way stopcock.
10. Three-way stopcock to attach several aniline water traps to react C<sup>14</sup> phosgene and convert it to diphenylurea.
- 11-14. Cooling traps to retain excess phosgene and methylene chloride evaporated from (5).
15. Stock valve.

FIGURE 1. APPARATUS FOR PREPARATION OF LABELED TOLUENE DIISOCYANATE

Specific activity of the TDI was determined in two ways. One was by preparing very thin counting samples of solid material obtained by reacting TDI with di-n-butylamine and evaporating the solvent. The other was by reacting TDI with  $\text{Ba}(\text{OH})_2$  solution and counting the resulting  $\text{BaCO}_3$  in an "infinitely thick" layer. In both cases adequate samples of standardized  $\text{BaC}^{14}\text{O}_3$  were used as standards. The results for the specific activity of TDI were in agreement within 20%.

#### Mixing and Curing of Labeled Propellant

The Aerojet-General Polaris urethane propellant formulation ANP 2639 AF was used. The apparatus used for mixing the radioactive propellant is shown in Figure 2. The entire propellant mixing procedure was carried out under a pressure of 10 mm Hg. When material had to be added the mixer was flushed with prepurified nitrogen. Gases evolved on mixing, including  $\text{C}^{14}\text{O}_2$ , were caught in the cooling traps (liquid nitrogen) of the apparatus. All ingredients used were carefully dried. The operation was started by mixing a premix of PPG, MDTA, and lecithin for 10 minutes. Coarse and fine ammonium perchlorate (blended together) was added in three equal portions, the mixing time after each addition being 10 minutes. In the next step, a premix of aluminum, carbon black, copper chromite, and phenyl beta-naphthylamine was added in two equal portions; mixing time again was 10 minutes after introducing each portion. The TDI (labeled diluted with unlabeled) together with ferric acetyl acetone were added; the total batch was mixed for 25 minutes. The mixture was cast in an aluminum cylinder and cured for 4 days at 43° C. Two batches of labeled propellant weighing 250 g each were prepared.

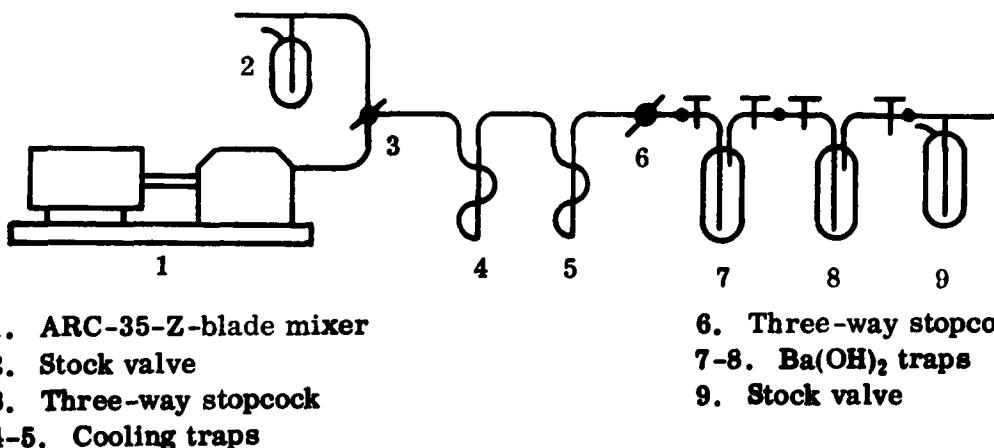


FIGURE 2. APPARATUS FOR MIXING OF LABELED PROPELLANT

In the first batch, an activity of 44 microcuries of TDI was incorporated. During curing the temperature went out of control, up to at least 130° C. This is thought to be the reason the propellant did not cure completely. Therefore, this batch was used only for a qualitative study.

In the second batch, 34 microcuries of labeled TDI was included. After 4 days of curing, a good rubbery propellant was obtained.

#### Counting of C<sup>14</sup>O<sub>2</sub> Evolved from Propellant

Radioactive carbon dioxide was expected to be evolved on mixing (because of traces of moisture causing hydrolysis of the TDI) and on storage of the propellant as a result of decomposition of the urethane linkages.

##### C<sup>14</sup>O<sub>2</sub> Evolved on Mixing:

The CO<sub>2</sub> evolved on mixing was condensed with liquid nitrogen, reacted with Ba(OH)<sub>2</sub> solution to form BaCO<sub>3</sub>, and finally the BaCO<sub>3</sub> counted with a Geiger-Mueller Counter after usual sample preparation (as an "infinitely thick" BaCO<sub>3</sub> layer).

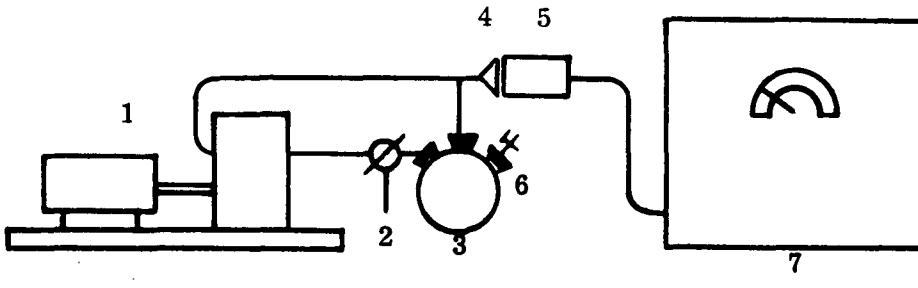
##### C<sup>14</sup>O<sub>2</sub> Evolved on Storage:

Continuously Working Counting System: The first approach was to use a continuously working counting system as shown in Figure 3 and similar to one described by Basolo.<sup>(9)</sup> The propellant sample is contained in flask 3. The gas over the propellant is circulated in a closed system by means of a pump (1). It passes a counting window (4) made of a very thin Mylar film, which allows the C<sup>14</sup> beta-radiation to penetrate into the Geiger-Mueller Counter (5) directly attached to the counting window. The counts are registered in the scaler (7). The stopcocks (2) and (6) serve to change the atmosphere in the closed system, if necessary.

The results obtained on testing this system showed that its counting efficiency is too low for the activities of the CO<sub>2</sub> evolved from the propellant sample. Therefore, a second technique of counting the activity was chosen.

Noncontinuous Counting System: This second method is based on a non-continuous counting procedure. The propellant sample is stored in a 100-ml flask with an outlet closed by means of a three-way stopcock. Through this three-way stopcock, the flask containing the sample is first evacuated and then filled with inert gas containing a certain amount of inactive CO<sub>2</sub> (approximately 10 ml CO<sub>2</sub>) which serves as carrier for the CO<sub>2</sub> evolved from the propellant

sample. In this way it is possible to sample even the very small amounts of CO<sub>2</sub> coming from the propellant because they mix with the added carrier.



- |                    |                           |
|--------------------|---------------------------|
| 1. Pump            | 5. Geiger-Mueller counter |
| 2. Stopcock        | 6. Stopcock               |
| 3. Flask           | 7. Scaler                 |
| 4. Counting window |                           |

FIGURE 3. COUNTING OF LABELED PROPELLANT

From time to time the storage flask is evacuated; all the CO<sub>2</sub> contained is trapped with liquid nitrogen and reacted with Ba(OH)<sub>2</sub> solution. The precipitated BaCO<sub>3</sub> is then prepared as usual for counting with a Geiger-Mueller Counter (as an "infinitely thick" layer). This method, as described, worked satisfactorily, the counting efficiency being high enough for the counted activities.

The activity of the BaCO<sub>3</sub> obtained from the samples was compared to standardized BaC<sup>14</sup>O<sub>3</sub> for calculating the specific and absolute activity.

## RESULTS

### Qualitative Results

#### Evolution of C<sup>14</sup>O<sub>2</sub> on Mixing:

The counting of the gases evolved on mixing after treating showed that the BaCO<sub>3</sub> obtained was radioactive. This indicates that TDI is decomposed on mixing. No quantitative investigation of this result has been done so far.

#### Evolution of C<sup>14</sup>O<sub>2</sub> on Storage:

The BaCO<sub>3</sub> samples obtained from the propellant on storage were found to be radioactive. Because the carbon atom of the urethane linkage of the propellant is the only site labeled, this indicates that the urethane linkage is decomposed on storage of the propellant. It is therefore a source for the CO<sub>2</sub> evolved on storage as reported. (6)

Checking of Continuous Counting System:

The continuous counting system was checked with a specimen of the unsatisfactorily cured propellant (approximately 125 g of batch no. 1, containing 22 microcuries of C<sup>14</sup>-labeled TDI). The entire propellant sample (a viscous slurry) was stored under air at 77° C. The results of this experiment are plotted in Figure 4. The counting rates obtained were rather low; and, because of this low counting efficiency, the system was not used for other experiments. Figure 4 also shows that evolution of CO<sub>2</sub> from the uncured propellant is rapid at the beginning, while it slows markedly to a steady rate after 3 days of storage.

Quantitative Results

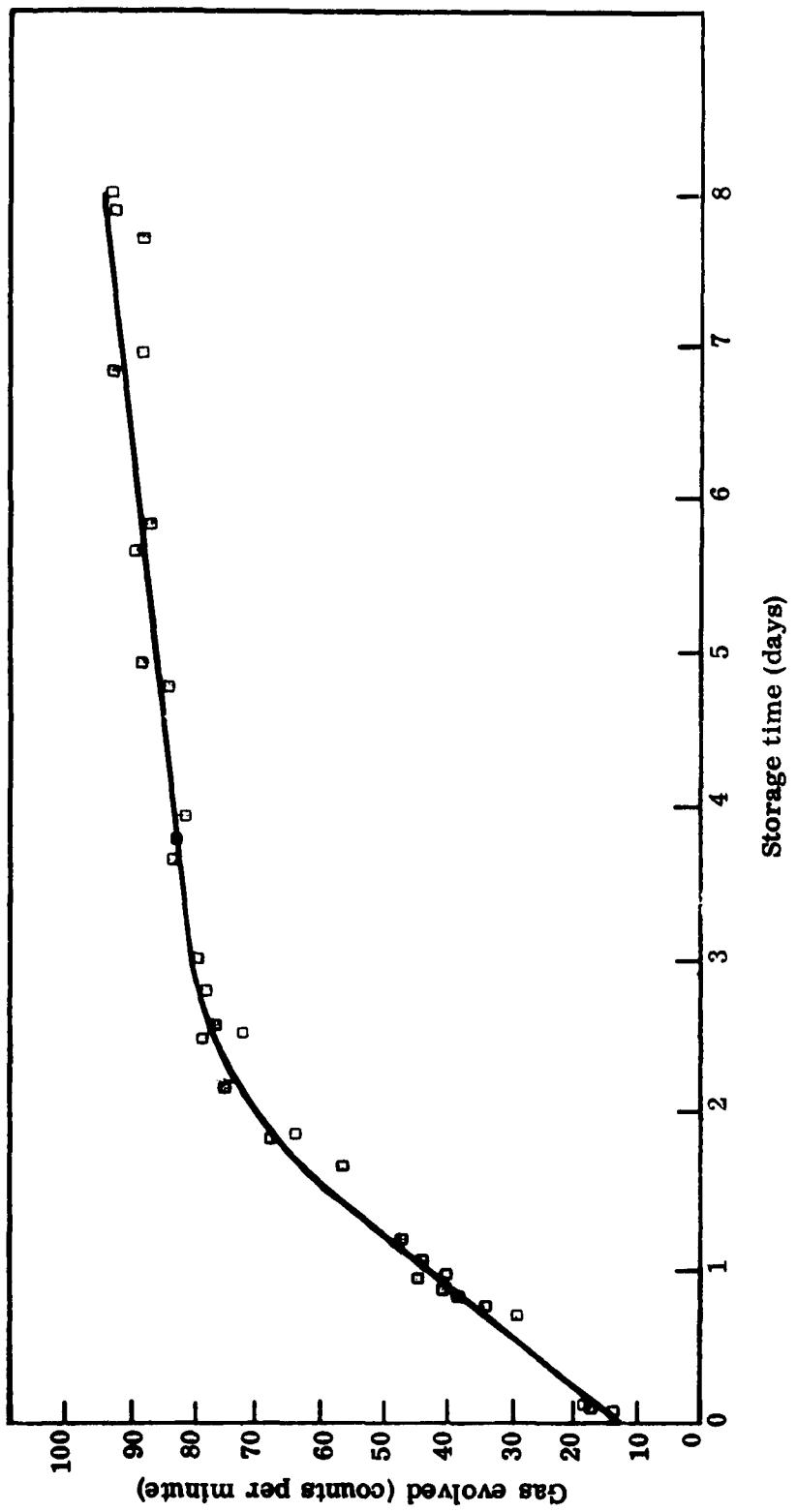
The noncontinuous counting procedure was used to investigate the CO<sub>2</sub> evolution rate from the radioactive propellant (batch no. 2).

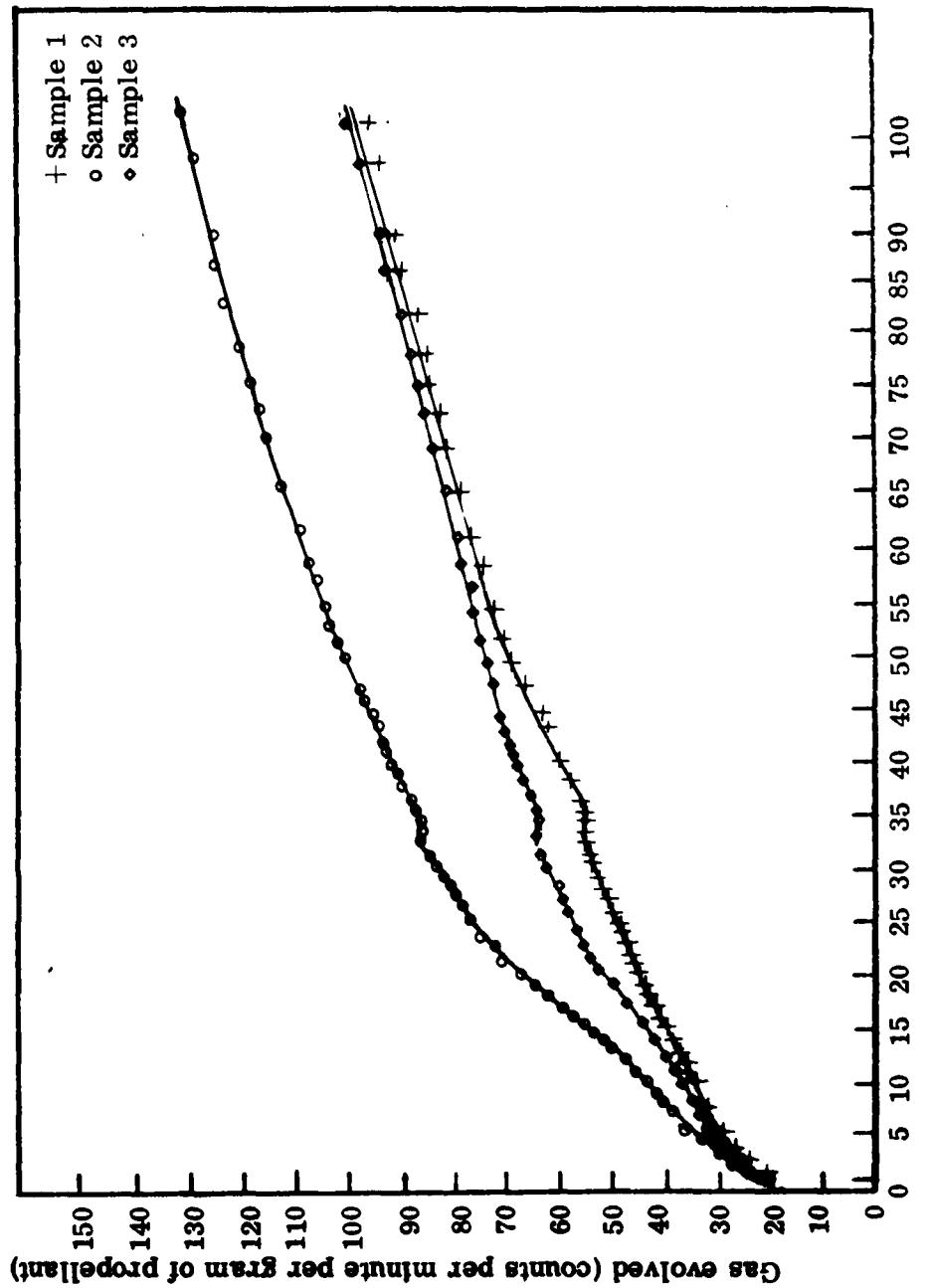
The propellant (169.3 g), containing 23.1 microcuries of TDI was cut in rectangles (1/4 × 1/2 × 3/4 inch) and stored in three equal portions in 100-ml flasks. An inert atmosphere of 40 ml nitrogen and 10 ml carbon dioxide was used, thus maintaining a pressure of about 0.5 atm in the storage flasks. The storage temperature was maintained at 71° C. Figure 5 shows the results obtained.

As shown in Figure 5, the CO<sub>2</sub> evolution rate becomes steady at about the 50th day of storage. The flat point in the curve between the 33rd and 35th day is caused by a breakdown of the heating system. It was found that the heating system was not completely adjusted at the beginning, causing a temperature difference of 3° C between Samples 1 and 2 and a temperature difference of about 1° C between Samples 1 and 3. This is thought to be the cause of the different rates of CO<sub>2</sub> evolution among the three samples. The difference in the evolution rate disappeared after some time. From the 60th day on, the CO<sub>2</sub> is evolved at an activity rate of 0.75 count per minute per day per gram of propellant.

Further, the following calculations can be made: 34.2 microcuries of C<sup>14</sup> as labeled TDI was incorporated in the original 250-g propellant mix; 169.3 g of propellant, containing 23.1 microcuries of labeled TDI, was recovered for storage. Therefore, 1 gram of propellant contained 0.14 microcurie of C<sup>14</sup>-TDI.

If the 75th day of storage is taken then there is an accumulated average (over all three samples) of 100 counts per minute per gram of propellant. This is a total activity of  $4 \times 10^{-3}$  microcuries (converted from counts per minute to microcuries by means of BaC<sup>14</sup>O<sub>3</sub> standard).

FIGURE 4.  $\text{CO}_2$  EVOLUTION FROM A-1 POLARIS PROPELLANT

FIGURE 5. EVOLUTION OF RADIOACTIVE CO<sub>2</sub>

As indicated above, at the beginning of storage each gram of propellant contained  $1.4 \times 10^{-1}$  microcuries of C<sup>14</sup>-TDI. Until the 75th day,  $4 \times 10^{-3}$  microcuries or 2.86% of this initial activity evolved from the sample as CO<sub>2</sub>. As every C<sup>14</sup>O<sub>2</sub>-molecule urethane linkage and as the other (C<sup>12</sup>) urethane linkages behave the same way, this means also that 2.86% of all the urethane linkages of the binder have been cleaved. If it is now assumed that the CO<sub>2</sub> evolution rate remains constant at 0.75 count per minute per day per gram of propellant (from the 60th day), then the useful life or half life of the propellant can be roughly predicted.

A C<sup>14</sup> activity evolved at a rate of 0.75 count per minute per gram per day means  $3 \times 10^{-5}$  microcuries per gram per day or 0.02% of the originally introduced activity. So if the rate stayed constant for the rest of the year, then the propellant binder would degrade 2.86% (first 75 days) plus 5.8% ( $0.02 \times 290$ ) which would equal 8.66% in the first year. Assuming constant rate, the following years would bring 7.3% degradation of the binder. Fifty percent of the binder would be decomposed after 6.7 years, thus the useful life or half life of the propellant is 6.7 years.

#### DISCUSSION

The experiments showed radioactive CO<sub>2</sub> is evolved on mixing, the only source of which is the labeled TDI. So far, no quantitative investigation has been made nor has the mechanism of the TDI decomposition been studied. It is believed, however, that the CO<sub>2</sub> evolution is from hydrolysis of the TDI caused by traces of moisture contained in the other propellant ingredients. It should be possible to investigate thoroughly the CO<sub>2</sub> evolution on mixing by this method.

This method should also be a valuable tool in investigating the CO<sub>2</sub> evolution on curing and furthering the optimal conditions of curing. Experiments have not yet been carried out.

It has been proven by the experiments performed in this study that cleavage of the urethane linkage of the propellant binder is one source for the CO<sub>2</sub> evolution observed on thermal decomposition of urethane propellants. The results do not at this point indicate whether this is the only source for CO<sub>2</sub> evolution; further experiments will be required to investigate this. The mechanism of the cleavage of the urethane linkage has not been determined yet. It is believed, however, that hydrolysis is not the only cause. The described tracer method is thought to be an effective tool to investigate the influence of such parameters as moisture, pressure, other ingredients of the propellant, etc., on the rate of CO<sub>2</sub> evolution and, therefore, rate of binder breakdown.

Furthermore, the method is thought to be of special value in predicting the stability and useful lifetime of a polyurethane propellant in advance. As the sensitivity of the method can be increased by a factor of  $10^4$  (by using a one hundred times higher C<sup>14</sup> activity and gas counting of the C<sup>14</sup>O<sub>2</sub>, which gives another factor of approximately  $10^2$ ), it should be possible to investigate the stability of polyurethane propellant binders at room temperature, a more realistic situation than the high temperature-accelerated cycle.

An additional advantage of the method is that only small amounts of propellant are required for surveillance. Additional experiments are required to determine broader applicability of the method.

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**U. S. Army**  
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